



## Polyaniline grafted melt-spun PET fibers for anionic dye removal

Nadia H. Elsayed<sup>a,b,\*</sup>, Raedah A.S. Alatawi<sup>c</sup>, Ohud H.N. Alhawiti<sup>a</sup>, I.S. Elashmawi<sup>d</sup>,  
Ayshah Alatawi<sup>e</sup>, Abdulhadi Hamad Al-Marri<sup>a</sup>

<sup>a</sup>Department of Chemistry, University College-Alwajh, University of Tabuk, Tabuk 71421, Saudi Arabia, emails: [nhussein@ut.edu.sa](mailto:nhussein@ut.edu.sa) (N.H. Elsayed), [oalhowiti@ut.edu.sa](mailto:oalhowiti@ut.edu.sa) (O.H.N. Alhawiti), [aalmarri@ut.edu.sa](mailto:aalmarri@ut.edu.sa) (A.H. Al-Marri)

<sup>b</sup>Polymers and Pigments Department, National Research Centre, 12622 Dokki, Giza, Egypt

<sup>c</sup>Department of Chemistry, Faculty of Science, University of Tabuk, Tabuk 71421, Saudi Arabia, email: [r.alatawi@ut.edu.sa](mailto:r.alatawi@ut.edu.sa) (R.A.S. Alatawi)

<sup>d</sup>Department of Spectroscopy, Physics Division, National Research Centre, Giza, Egypt, email: [islam\\_shukri2000@yahoo.com](mailto:islam_shukri2000@yahoo.com) (I.S. Elashmawi)

<sup>e</sup>Physics Department, Faculty of Science, University of Tabuk, Tabuk 71421, Saudi Arabia, email: [ase.alatawi@ut.edu.sa](mailto:ase.alatawi@ut.edu.sa) (A. Alatawi)

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### ABSTRACT

Polyethylene terephthalate (PET) fibers were successfully prepared via the melt spinning method and modified by oxidative grafting polymerization of aniline monomer on the fiber surface in two different acidic media namely hydrochloric and sulphuric acid. The produced polyaniline grafted PET acquired improvement in its morphological properties and used as a pollutant sorbent for the removal of anionic direct dye namely Direct Violet 93 from aqueous solutions. Fibers of pure PET and polyaniline grafted PET in sulphuric acid was characterized using Fourier-transform infrared spectroscopy, scanning electron microscopy, and X-ray diffraction measurements. The dye removal was studied under different conditions including aniline monomer and initial dye concentrations. The results indicated that using sulphuric acid is more effective than hydrochloric acid for grafting of polyaniline onto PET fiber surface and for dye removal.

*Keywords:* Direct dyes; Dye removal; Wastewater treatment; Polyaniline; Polyester

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### 1. Introduction

Polyethylene terephthalate (PET) is considered one of the most common polymers which can be converted into fibers via the melt spinning process [1,2]. The produced PET fibers have unique mechanical and bulk properties. PET melt-spun fibers have different applications in many fields including construction, packaging, and water treatment [3–5]. Physical and chemical properties of the melt-spun PET fibers could be enhanced by chemical modification of the fiber surface via grafting polymerization of monomers onto the PET fiber surface [5]. Grafting of polyaniline onto the PET surface is one of the most important methods to

improve the conductivity of the melt-spun PET fibers due to the low cost, chemical stability, good electrical properties, and ease polymerization of aniline monomer [6,7].

During the past decade, dye removal is one of the most important routes for industrial wastewater treatment. The dye can be removed from wastewater through different methods as membrane filtration [8], photocatalytic decomposition [9,10], and adsorption [11,12]. Adsorption technique seems to be the most effective method for dye removal due to its high adequacy and low pollution bad effects [13,14].

This work aims to produce PET melt-spun fibers via melt spinning technique followed by modification of the produced fibers by grafting of polyaniline on fiber surface

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\* Corresponding author.

using in different acidic media using a chemical oxidative polymerization method. The produced surface grafted PET melt-spun fibers were used in the adsorption of direct anionic dye from the water solution.

## 2. Experimental setup

### 2.1. Materials

Commercial homopolymer PET granules were provided by SABIC (KSA). The PET-HC100 is characterized by an intrinsic viscosity (IV) of 0.84 dl/g, melting range between 240°C and 260°C and a crystalline density <1,400 kg/m<sup>3</sup>. Aniline 99% (Loba Chemie Pvt. Ltd., India) was purified by distillation before use. Potassium persulfate (PPS), hydrochloric acid (HCl), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), were obtained from Sigma-Aldrich, Germany. Cuprophenyl Violet 230 and Direct Violet 93 (DV93) was obtained from Ciba-Geigy (Switzerland).

### 2.2. Production of melt-spun PET fiber [15]

In order to prepare melt-spun PET fiber via the melt spinning process, PET granules were dried at 90°C for 24 h in a vacuum oven. Fiber melt spinning was carried out under the melt spinning conditions listed in Table 1 using a spinning apparatus (HAAKE single screw extruder, Germany) consisting of an extruder and a gear pump to control precisely the throughput rate, which was kept at 40 g/min for all polymers. Filaments extruded through a 24-hole spinneret with a diameter of 0.3 mm and at 275°C were taken up by the high-speed winder SW46 of BARMAG Company (China) below the spinneret.

### 2.3. Grafting of polyaniline (PAn) onto PET melt-spun fiber [16]

Grafting of polyaniline onto the produced PET melt-spun fiber was carried out by chemical oxidative polymerization method in a polymer tube in two different acidic media 1.0 M HCl or 1.0 M H<sub>2</sub>SO<sub>4</sub> according to the molar ratio 1:0.9 for monomer (aniline) and an oxidizing agent (PPS) respectively. The melt-spun PET fiber soaked in an aqueous solution of 1.0 M HCl or 1.0 M H<sub>2</sub>SO<sub>4</sub> for 30 min and exposed to ultrasonic treatment for 20 min under 300 W to improve

the diffusion of the oxidizing agent into the melt-spun fiber. Aniline solution (0.05 M: 0.5 M) was added into the reaction mixture rapidly for grafting in the shaker water bath at 30°C for 30 min. A green precipitate was deposited on the fabrics; the excess polymer was removed from the fiber by washing with distilled water and dried in an oven at 70°C for 3 h to produce (PAn-HCl/PET) and (PAn-H<sub>2</sub>SO<sub>4</sub>/PET).

The grafting percentage was determined by weighing fabric substrates before and after polymerization and using the following equation [17]:

$$\text{Grafting percentage (\%)} = \frac{(\text{grafted fiber weight}) - (\text{pure fiber weight})}{(\text{pure fiber weight})} \times 100 \quad (1)$$

### 2.4. Instruments

X-ray diffraction (XRD) measurements were performed using a Philips powder diffractogram PW 1050 (Germany) with ADM software and with Ni-filtered Cu-K radiation. The accelerating voltage was 40 kV, and the current was 30 mA. The morphology and fracture surface of the polymer and its composite were examined by scanning electron microscopy (SEM) analysis using a Zeiss DSM 962 microscope (Japan).

The Fourier-transform infrared spectroscopy (FTIR) were obtained using a Bruker IFS 28 spectrometer 67 (USA).

The residual concentration of the dye in the solution was analyzed and measured using Perkin Elmer UV-Vis Spectrophotometer (USA) at its maximum absorption wavelength (520 nm).

### 2.5. Using of PET melt-spun and polyaniline grafted PET melt-spun fiber in adsorption of anionic dye

For the dye adsorption experiments, batch mode studies were conducted using 0.1 g (±0.01) of PET melt-spun (PET) and polyaniline (PAn) grafted PET melt-spun fiber in both acidic media (PAn-HCl/PET) and (PAn-H<sub>2</sub>SO<sub>4</sub>/PET) taken separately as adsorbent. Each adsorbent was shaken in a 10 ml aqueous solution of (0.1:0.5 g/L) of DV93 anionic dye at a constant speed of 160 rpm at room temperature for 30 min adsorption time. At the end of the pre-determined time intervals, the adsorbent was removed. The absorbencies of samples were measured at λ<sub>max</sub> = 520 nm. Then the concentrations of the samples were determined by using a calibration curve. The adsorbed amounts of dye onto polyaniline were calculated by subtracting the amount taken by the fabric only from the amount taken by the grafted PET. Most of the experiments were carried out in triplicate at each condition where mean values are presented.

The adsorbed amount of dye was calculated using the equation [18]:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

where C<sub>0</sub> and C<sub>e</sub> are the initial and equilibrium concentrations of dye (mg/L), m is the mass of adsorbent dose (g), q<sub>e</sub> in mg/g is the amount of the adsorbed dye and V is the volume of solution (L).

Table 1  
Melt spinning conditions of PET melt-spun fiber

Temperature	PET
Zone 1°C	280
Zone 2°C	280
Zone 3°C	285
Pump, °C	290
Spinneret, °C	289
Melt, °C	292
Spinneret	
Pressure, bar	42
Winding speed	
Winder, m/min	3,000

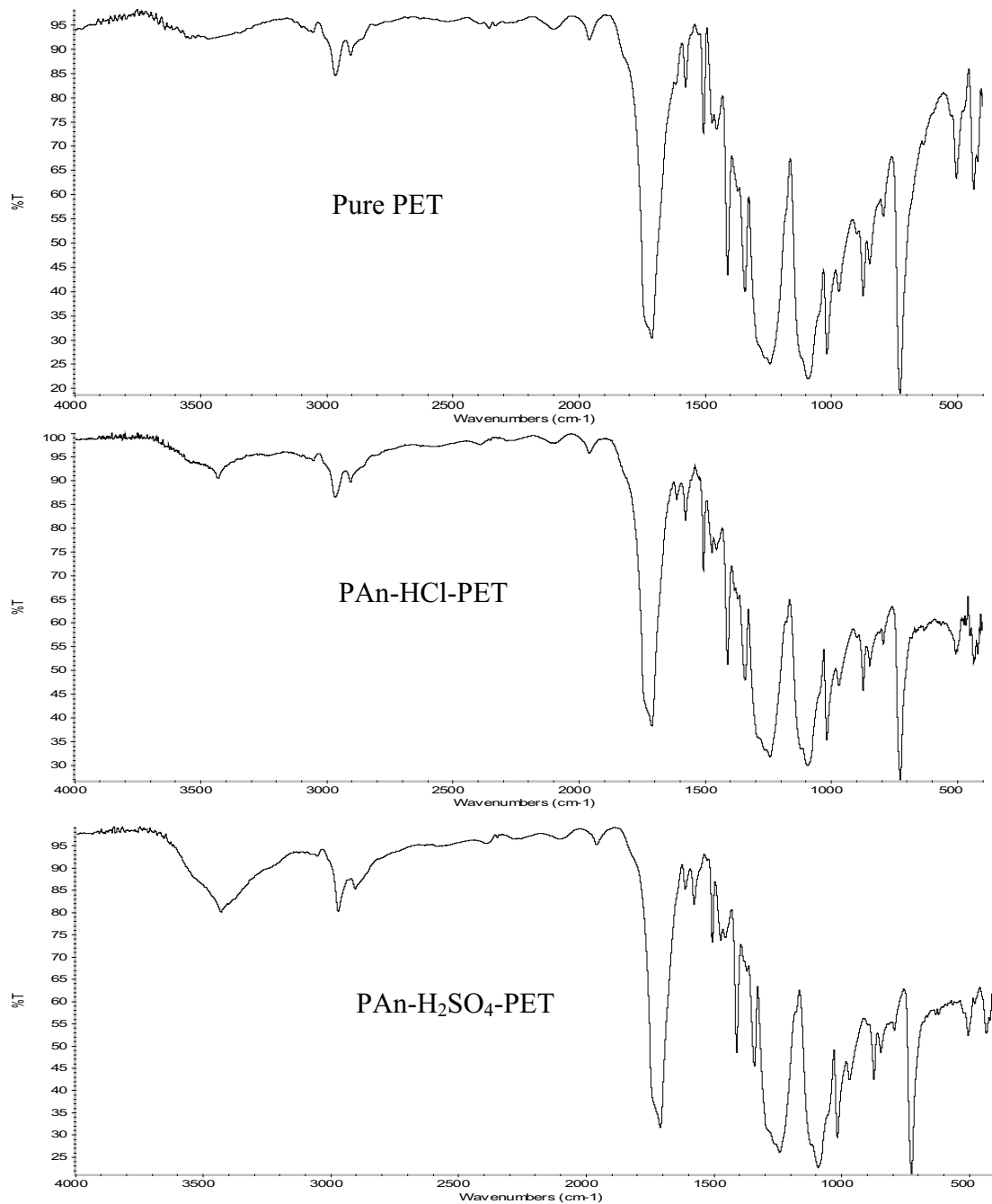


Fig. 1. FTIR spectrum of (a) pure PET, (b) PAn-HCl/PET, and (c) PAn-H<sub>2</sub>SO<sub>4</sub>/PET.

### 3. Results and discussion

#### 3.1. Characterization of PAn grafted PET

##### 3.1.1. Fourier-transform infrared spectroscopy

Fig. 1 explains FTIR spectra of pure PET melt-spun fiber and grafted PET with PAn in the presence of two different acids (HCl and H<sub>2</sub>SO<sub>4</sub>) are illustrated in Fig. 1. From the figure, it is evident that the PET fiber has characteristic bands at 2,970; 1,710; 1,243; and 725 cm<sup>-1</sup> which are assigned to C–H stretching, C=O stretching, C–O stretching and out of plane

bending of the *p*-disubstituted benzene ring in polyester respectively [19,20]. For grafted PET with PAn, the characteristic band around 3,430 cm<sup>-1</sup> is due to NH stretching of aromatic amine, 1,613 cm<sup>-1</sup> is due to C=C stretching vibration of quinoid structure and 1,506 cm<sup>-1</sup> is related to benzenoid rings in PAn. Additionally there are two characteristic small peaks overlapped with PET bands at 1,290 and 1,175 cm<sup>-1</sup> assigned to the C–N and C=N stretching vibrations respectively. Regarding the type of the used acidic medium in the grafting process, it is obvious that there is no difference in the bands related to the variation of the acid anions Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> [21].

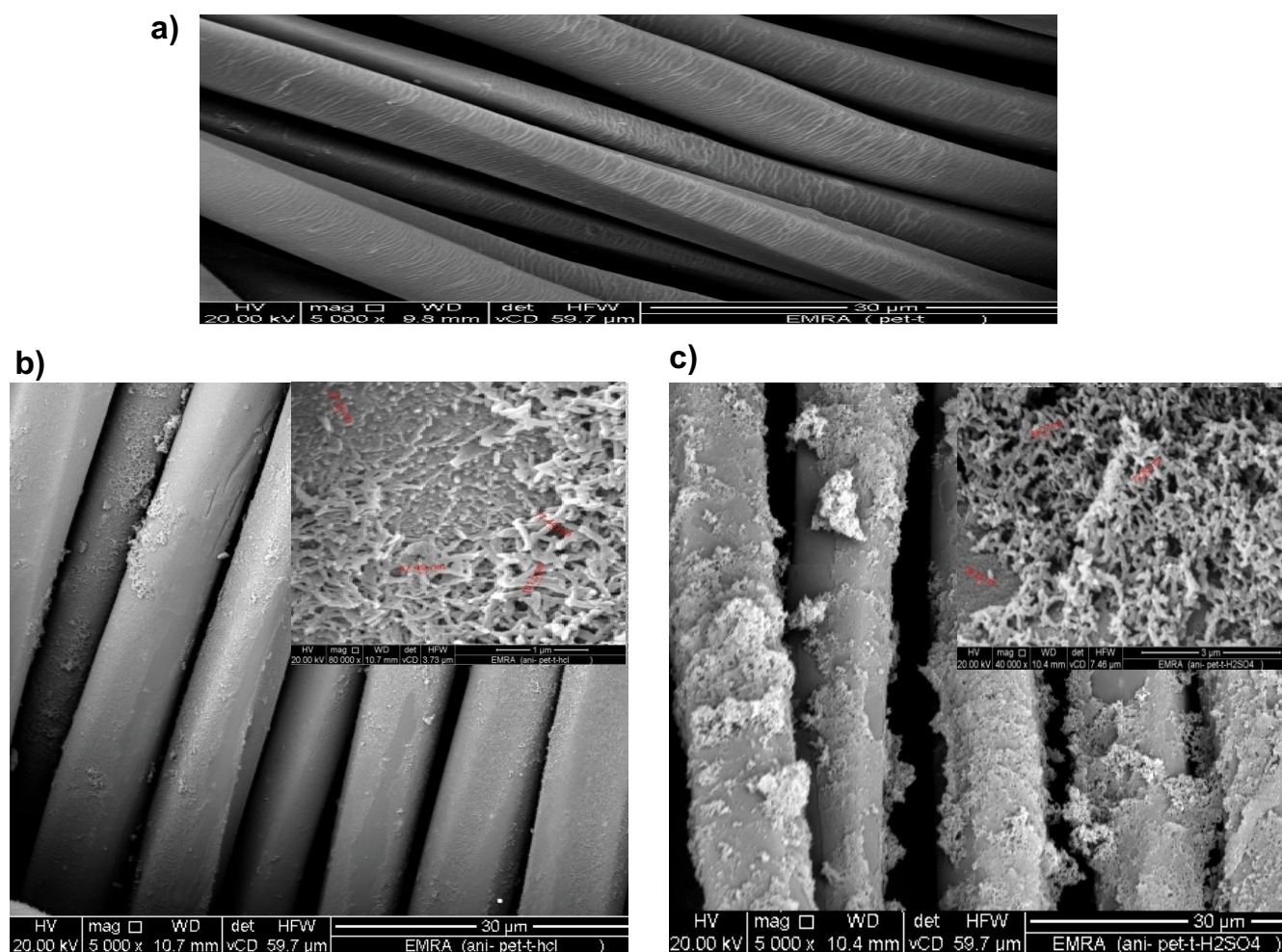


Fig. 2. SEM images of chemically synthesized PAN in different acids, [aniline] = 0.3 M, [PPS] = 0.09 M [HCl] and  $[H_2SO_4] = 1.0$  M. (a) Pure PET, (b) PAN-HCl/PET, and (c) PAN- $H_2SO_4$ /PET.

### 3.1.2. Scanning electron microscopy

Fig. 2 shows SEM images of pure and grafted PET with PAN at two different acidic media. The grafted samples showed network nanostructures or nanofibers form [22,23]. Comparing the images obtained using two different acids, PAN- $H_2SO_4$ /PET is more beneficial for dye adsorption.

### 3.1.3. X-ray diffraction

Sharp peaks refer to crystalline regions while broad ones refer to the amorphous region in the XRD pattern of most polymers [24]. Fig. 3 shows the XRD pattern of pure PET and (PAN- $H_2SO_4$ /PET) melt-spun fibers. The figure shows three diffraction peaks for pure PET fiber corresponding to  $2\theta$  values of  $18^\circ$ ,  $23^\circ$ , and  $26^\circ$  [25].

Comparing to XRD of (PAN- $H_2SO_4$ /PET), it is obvious that, the intensity of PET peaks decreased and slightly broadened after grafting with PAN in the presence of  $H_2SO_4$  which could explain the disturbance in the crystalline region of the fabric material due to grafting process. These results predict increasing in the dye absorption by using (PAN- $H_2SO_4$ /PET) more than using pure PET as dye absorbent because

the decrease in fiber crystalline led to an increase in fiber surface area and then increase in dye absorption [26,27].

## 3.2. Direct dyes uptake using PAN grafted PET

Two different adsorbents namely pure PET and PAN- $H_2SO_4$ /PET have been used in adsorption of anionic dye namely DV93 from aqueous solution. The effect of aniline monomer concentration and initial dye concentration on the amount of adsorbed dye was studied. The amount of adsorbed dyes relative to the amount of used PET fiber and the amount of grafted polyaniline was calculated and expressed in mg dye/g PET and mg dye/g PAN.

### 3.2.1. Effect of aniline monomer concentration

The effect of aniline concentration on the amount of adsorbed DV93 dye relative to the amount of used PET fiber and amount of grafted polyaniline by pure PET and PAN- $H_2SO_4$ /PET at adsorption time (30 min) and initial dye concentration 0.5 g/L at room temperature was studied and the resulted data is listed in Table 2. The dye adsorbed was

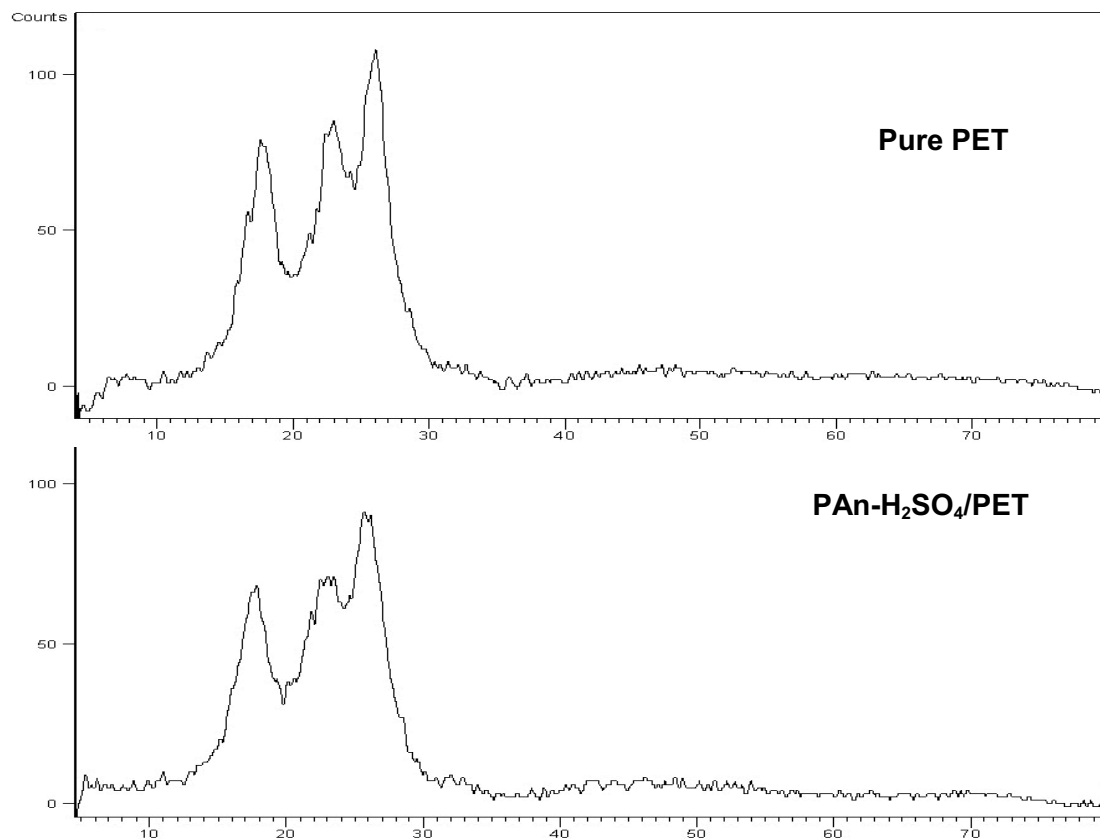


Fig. 3. X-ray diffraction patterns of (a) pure PET and (b) PAn-H<sub>2</sub>SO<sub>4</sub>-PET, [aniline] = 0.3 M, [PPS] = 0.09 M and [H<sub>2</sub>SO<sub>4</sub>] = 1.0 M.

Table 2

Effect of aniline concentration on the amount of adsorbed DV93 using pure PET and PAn-H<sub>2</sub>SO<sub>4</sub>-PET, [DV93] = 0.5 g L<sup>-1</sup> and the adsorption time = 30 min

[Aniline]	Sample	Graft yield %	Amount of adsorbed dye	
			mg dye/g PET	mg dye/g PAn
–	PET	–	12.4	–
0.05 M	PAn/H <sub>2</sub> SO <sub>4</sub> /PET	2.24	19.5	197
0.1 M	PAn/H <sub>2</sub> SO <sub>4</sub> /PET	4.06	22.7	310
0.3 M	PAn/H <sub>2</sub> SO <sub>4</sub> /PET	13.17	29.4	308
0.5 M	PAn/H <sub>2</sub> SO <sub>4</sub> /PET	20.58	29.5	311

calculated related to two different indicators, the amount of adsorbed dye per gram of PET and the amount of adsorbed dye per gram of grafted polyaniline. From the data it is clear that, the amount of adsorbed dye per gram of PET is markedly increased by increasing aniline concentration from 0.05 to 0.3 M afterward it becomes constant. Most probably, at the beginning more adsorbent surface area is created and more adsorption sites are available and consequently considerable dye amount adsorbed [28]. Increasing aniline concentration above 0.3 M would certainly increase the graft yield on PET significantly causing aggregations of active functional groups of PAn, that's the reason behind the plateau obtained.

From the other hand, the amount of adsorbed dye per gram of grafted polyaniline is markedly increased by increasing aniline concentration until 0.1 M aniline afterwards it becomes constant.

### 3.2.2. Effect of initial dye concentration

The effect of initial dye concentration of DV93 on both the amount of adsorbed dye per gram of PET per amount of grafted polyaniline was studied and the result data is presented in Table 3. The experiment was conducted with 0.1 g of PAn-H<sub>2</sub>SO<sub>4</sub>/PET added to 10 ml of different initial DV93 concentrations (0.1, 0.2, 0.4, and 0.5 mg/L) for

30 min. From the data, it is clear that, the amount of the dye adsorbed increased by increasing the initial dye concentration of solution up to the concentration 0.4 mg/g then it decreased again due to an increase in the driving force of the concentration gradient with the higher initial dye concentration.

From the data, it is also clear that, the efficiency is inversely proportional to initial dye concentration due to the increase of the residual concentration of dye molecules with increasing the initial dye concentrations. In other words, at higher concentrations of the dyes, the dye was not completely adsorbed, indicating that there is a saturation limit for the polymer above which it does not remove the dyes [29]. In the case of lower concentrations, the ratio of the initial number of dye molecules to the available adsorption sites is low and subsequently the fractional adsorption becomes independent of initial concentration [30].

The data in Table 3 indicated also to the amount of adsorbed dye per gram of grafted polyaniline and it is obvious that, the amount of dye is markedly increased by increasing initial dye concentration until 0.4 g/L and then it decreased again aggregations of active function groups of PAN.

#### 4. Conclusion

In this study polyaniline was grafted onto melt-spun PET in the presence of two different types of acidic medium (HCl and H<sub>2</sub>SO<sub>4</sub>) using oxidative grafting polymerization method and the resulted fibers were characterized by FTIR, SEM, and XRD, then used as sorbent material for DV93 anionic dye removal from water solution. It was found also that the dye adsorption using PAN-H<sub>2</sub>SO<sub>4</sub>/PET for DV93

increased by increasing the initial aniline and dye concentration up to 0.3 M aniline concentration and 0.5 g/L initial dye concentration.

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Table 3

Effect of initial dye concentration of on the amount of adsorbed DV93 dye using pure PET and PAN-H<sub>2</sub>SO<sub>4</sub>/PET, [aniline] = 0.3 M and the adsorption time = 30 min at room temperature

[DV93]	Amount of adsorbed dye	
	mg dye/g PET	mg dye/g PAN
0.01 g/L	–	–
	0.75	–
	2.54	33
0.1 g/L	–	–
	3.5	–
	9.6	143
0.2 g/L	–	–
	8.1	–
	18.7	240
0.4 g/L	–	–
	14.9	–
	33.1	355
0.5 g/L	–	–
	11.8	–
	29.4	308

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